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<p><b>Risk Reduction and Environmental Stewardship— Remediation Program</b></p> <p><b>Standard Operating Procedure</b></p> <p>for <b>Routine Validation of Volatile Organic Data</b></p>		
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## Revision Log

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# Routine Validation of Volatile Organic Data

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# Routine Validation of Volatile Organic Data

## 1.0 PURPOSE

- 1.1 This standard operating procedure (SOP) represents the minimum standards for evaluating routine volatile organic compound (VOC) analytical data. These data are generated for the Los Alamos National Laboratory (LANL), Risk Reduction and Environmental Stewardship—Remediation (RRES-R) Program, using SW-846 Method 8260 or the comparable Contract Laboratory Program (CLP) methods under the current statement of work (SOW) for analytical services. The evaluation of data by this procedure is not specific to a particular data use, although this procedure may be used to develop focused data-validation requirements specific to a particular data use.
- 1.2 Implementation of this procedure will result in a tabulation of data compliances and noncompliances identified relative to expectations for data quality based on national guidelines for data review (U.S. Environmental Protection Agency [EPA] 1999, 66649). Data noncompliance is noted through the application of qualifier flags (Attachment A) and reason codes (Attachment B) to the reported results. Because the acceptance criteria used for this procedure are not based on site-specific acceptance criteria, the results of this validation procedure are intended to be used as general indicators of data quality and should not be misconstrued as a definitive identification of data usability.
- 1.3 Nothing in this SOP precludes the validator from going beyond the minimum requirements specified in this SOP. To address data-quality issues in a data package, the **validator** may assign qualifiers based on his or her professional judgment.
- 1.4 Implementation of this procedure also may be followed by a more focused and data-use-specific evaluation of the data, especially if the implementation of this SOP indicates that the data may contain technical deficiencies. The **validator** shall note any need for a more focused validation on the Data-Validation Cover Sheet (Attachment C).
- 1.5 The **validator** shall use the VOC Data-Validation Checklist (Attachment D) to record the specific validation steps conducted.

## 2.0 SCOPE

- 2.1 All **RRES-R Personnel** shall implement this mandatory SOP when evaluating routine VOC analytical data for the RRES-R Program.
- 2.2 **Subcontractors** performing work under the RRES-R Program's quality program shall follow this SOP.

## 3.0 TRAINING

- 3.1 **RRES-R Personnel** shall train to and use the current version of this SOP; contact the author if the SOP text is unclear.
- 3.2 **RRES-R Personnel** using this SOP shall document training in the RRES-R training database located at <http://erinternal.lanl.gov/Training/login.asp> in accordance with QP-2.2.
- 3.3 The responsible **supervisor** shall monitor the proper implementation of this procedure and ensure that the appropriate personnel complete all applicable training assignments.
- 3.4 All **data validators** implementing this SOP shall possess a minimum of a bachelor's degree in chemistry or one of the physical sciences and either two years of experience in generating analytical data in an environmental analytical laboratory or two years of data-validation experience.
- 3.5 RRES-R **validators** shall review and sign inexperienced RRES-R Program validators' data-record packages until ten data-record packages produced according this data-validation SOP are satisfactorily validated.
- 3.6 RRES-R Program **validators** shall demonstrate familiarity with the EPA national functional guidelines for data review.

## 4.0 DEFINITIONS

- 4.1 *Analyte*—The element, nuclide, or ion that a chemical analysis seeks to identify and/or quantify; the chemical constituent of interest.
- 4.2 *Area count*—The Integrated area under a chromatographic peak. The area count is proportional to the amount of compound present in the aliquot introduced into the chromatograph.
- 4.3 *Continuing calibration verification (CCV)*—The check standards used to determine if the instrument response to analyte concentration is within acceptable bounds relative to the initial calibration. A CCV is performed after every 12 h of an operation or (for inorganics and high explosives [HEs]) after every 10 injections (samples and/or quality control [QC] samples), whichever is more frequent, thus verifying the satisfactory performance of an instrument. The continuing calibration 12-h period

assumes that the instrument has not been shut down since the initial calibration.

- 4.4 *Data validator*—The person who has met the minimum standards of training established by the RRES-R Program for data-validation and who performs data-validation on behalf of the RRES-R Program (hereafter referred to as the “validator”).
- 4.5 *Detect (inorganic and organic)*—The sample result above the method detection limit (MDL) reported by the contract analytical laboratory. The contract laboratory reports the concentration of the analyte in the sample.
- 4.6 *Form 1*—The Organic analysis data sheet for each individual sample that includes information needed to identify the sample and the analytical results for the sample. See the SOW for analytical services (RFP No. 9-XS1-Q4257) for a more complete definition.
- 4.7 *Holding time*—The maximum elapse of time that a sample can be stored without unacceptable changes in analyte concentrations. Holding times apply under prescribed conditions, deviations from these conditions may affect the holding time. Extraction holding time refers to the time lapse from sample collection to sample preparation; analytical holding time refers to the time lapse between sample preparation and analysis.
- 4.8 *Initial calibration*— The process used to establish the relationship between instrument response and analyte concentration at several analyte-concentration values to demonstrate that an instrument is capable of acceptable analytical performance.
- 4.9 *Instrument performance check*—An analysis of a chemical of known relative mass abundance that indicates how well a mass spectrometer is calibrated.
- 4.10 *Internal standard (IS)*—The chemical compound added to every blank, sample, and standard extract at a known concentration that is used to (1) compensate for analyte concentration changes that might occur during storage of the extract and (2) compensate for quantitation variations that can occur during analysis. ISs are used as the basis for quantitation of the target analytes.
- 4.11 *Laboratory control sample (LCS)*—A known matrix that has been spiked with compound(s) representative of the target analytes. The LCS is used to document laboratory performance. The acceptance criteria for LCSs are method specific.
- 4.12 *Laboratory duplicate sample*—The portions of a sample taken from the same sample container, prepared for analysis and analyzed independently but under identical conditions; used to assess or demonstrate acceptable

laboratory-method precision at the time of analysis. Each duplicate sample is equally representative of the original material. Duplicate analyses also are performed to generate data, and to determine the long-term precision of an analytical method on various matrices.

- 4.13 *Laboratory qualifier (or laboratory flag)*—The codes applied to the data by the contract analytical laboratory to indicate, on a gross scale, a verifiable or potential data deficiency. These flags are applied using the EPA CLP guidelines (EPA 1994, 48639; EPA 1999, 66649).
- 4.14 *LANL data-validation qualifiers*—The data qualifiers defined by LANL and used in the RRES-R Program routine-validation process. Attachment A lists all the data qualifiers that are applicable to the analytical suites.
- 4.15 *LANL data-validation reason codes*—The codes applied to the sample data by data validators who are independent of the contract laboratory that performed the sample analysis. Reason codes provide an in-depth and analysis-specific explanation for applying the qualifier along with a description of the potential impact on the data use. For a complete list of data qualifiers applicable to any particular analytical suite, consult the appropriate RRES-R Program SOP.
- 4.16 *Lower acceptance limit (LAL)*—The lowest limit that is acceptable based on the QC criteria for a specific QC sample for a specific method. Any results lower than the LAL are qualified following this routine validation procedure.
- 4.17 *Matrix spike*—An aliquot of sample spiked with a known concentration of the target analyte(s). Matrix-spike samples are used to measure the ability to recover prescribed analytes from a native sample matrix. Spiking typically occurs before sample preparation and analysis.
- 4.18 *Method blank*—An analyte-free matrix to which all reagents are added in the same volumes or proportions as those used in the environmental sample processing, and which is prepared and analyzed in the same manner as the corresponding environmental samples. A method blank is used to assess the potential for sample contamination during preparation and analysis.
- 4.19 *Method detection limit (MDL)*—The minimum concentration of a substance that can be measured and reported with known statistical confidence that the analyte concentration is greater than zero. The MDL is determined by analyzing samples of a given matrix type that contain the analyte after the sample is subjected to the usual preparation and analyses. The MDL is used to establish detection status.
- 4.20 *Nondetect (organics)*—The sample result that is less than the MDL. The laboratory reports nondetects as undetected at the reporting limit (RL).

- 4.21 *Percent difference (%D)*—The measure of the deviation from the initial calibration to the continuing calibration based on calibration factors.
- 4.22 *Percent recovery (%R)*—The amount of material detected in a sample (minus any amount already in the sample) divided by the amount added to the sample and expressed as a percentage.
- 4.23 *Percent relative standard deviation (%RSD)*—The evaluation of the deviation between the concentration versus analyte response over the dynamic linear calibration range. The basic equation is  $\%RSD = (\text{Std dev}/\text{av}) \cdot 100$ .
- 4.24 *Relative response factor (RRF)*—The relationship between analyte concentration versus area response.
- 4.25 *Reporting limit (RL)*—The lowest concentration that can be reliably achieved within specified limits of precision and accuracy during routine analytical-laboratory operating conditions. The low point on a calibration curve should reflect this RL. The RL is not used to establish detection status.
- 4.26 *Request number (RN)*—An identifying number assigned by the RRES-R Program to a group of samples that are being submitted for analysis.
- 4.27 *Routine data*—The data generated using analytical methods that are identified as routine methods in the current RRES-R Program SOW for analytical services.
- 4.28 *Routine data validation*—The process of reviewing analytical data relative to quantitative routine acceptance criteria. The objective of routine data validation is two-fold: (1) to estimate the technical quality of the data relative to minimum national guidelines adopted by the RRES-R Program; and (2) to indicate to data users the technical data quality at a general level by assigning qualifier flags to environmental data whose quality indicators do not meet the acceptance criteria.
- 4.29 *Surrogate compound (surrogate)*—An organic chemical compound used in the analyses of organic target analytes that is similar in composition and behavior to the target analytes but is not normally found in environmental samples. Surrogates are added to every blank, sample, and spike to evaluate the efficiency with which analytes are recovered during extraction and analysis.
- 4.30 *Target analyte*—An element, chemical, or parameter for which the concentration, mass, or magnitude is designed to be quantified by use of a particular test method.
- 4.31 *Tentatively identified compound (TIC)*—The chemical compound detected in a sample that is not a target analyte, internal standard (IS), or surrogate



compound. Up to 30 chromatographic peaks may be subject to mass spectral matching for identification as a TIC. Tentative identification is based on the comparison of the compound mass spectrum to an industry-standard mass-spectra library using both a statistical matching algorithm and the professional judgment of the analyst.

- 4.32 *Upper acceptance limit (UAL)*—The highest limit that is acceptable, based on the QC criteria for a specific QC sample for a specific method. All results greater than the UAL are qualified following this routine validation procedure.

## 5.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure:

- Data Validator (see definition 4.4)
- RRES-R Personnel
- Supervisor

## 6.0 PROCEDURE

**Data validators** shall perform the following work processes. Make any deviations from this SOP in accordance with QP-5.7 and/or SOP-01.01.

### 6.1 Preparing for Data Validation

1. Obtain the required current version of the VOC Data-Validation Checklist form (Attachment D) from the RRES-R Program website (<http://erinternal.lanl.gov/Quality/forms.htm>).
2. From the Sample Management Office (SMO), obtain the data-record packages that contain the sample data to be validated.
  - A. Prepare a Data-Validation Cover Sheet (Attachment C) by completing Section I of the cover sheet and placing a check or other mark adjacent to the analytical suites for which this validation is being performed.
  - B. If any data are rejected, check the rejected box and notify the project chemist immediately.

**Note:** You may use a single cover sheet when validating multiple analytical suites under the same requestor number (RN).

**Note:** Use a separate sheet of paper to document each deficiency identified beyond the extent of this procedure, including phone conversations with the analytical laboratory concerning these

deficiencies. Attach these sheets to the Data-Validation Cover Sheet.

3. Verify that the following items are present in the data-record package:

- The signed LANL COC record
- The case narrative
- The result forms (CLP Form 1 or equivalent) for each sample
- The reconstructed ion chromatograms (RICs) for each sample
- The RICs for standards
- The raw and background subtracted spectra (BSS) of identified compounds
- The quantitation reports
- The QC forms (CLP 2A [Deuterated Monitoring Compound Recovery], 3A [Matrix Spike/Matrix Spike Duplicate Recovery], 4A [Method Blank Summary], 6A [gas chromatography/mass spectrometryGC/MS] Initial Calibration Data], 8A [Internal Standard Area and retention time {RT} Summary], or equivalent) for water and/or soils, as appropriate
- The tentatively-identified compound (TIC) forms (CLP Form V-TIC, or equivalent), which are required only if the RRES-R Program requested a TIC report
- The mass spectra of the TICs with the three best library matches (required only if the RRES-R Program requests TIC reports)

4.

IF the required documentation for the data-record package is...	FOR...	THEN...
Complete,		<ul style="list-style-type: none"><li>• Go to Step 6.</li></ul>
Missing,	< 6 mo.	<ul style="list-style-type: none"><li>• Contact the analytical laboratory and/or the SMO.</li><li>• Allow 3 business days for submittal.</li><li>• Go to Step 5.</li></ul>

4.	<b>IF the required documentation for the data-record package is...</b>	<b>FOR...</b>	<b>THEN...</b>
	Missing,	= 6 mo.	<ul style="list-style-type: none"> <li>• Contact the analytical laboratory and/or the SMO.</li> <li>• Allow 10 business days for submittal.</li> <li>• Go to Step 5.</li> </ul>

**Note:** To expedite the validation process, the validator may request that the contract laboratory forward the missing information directly to the validator by fax or e-mail within 24 h of notification.

5.	<b>IF the analytical laboratory...</b>	<b>THEN...</b>
	Submits the documentation within the specified period of time,	<ul style="list-style-type: none"> <li>• Go to Step 6.</li> </ul>
	Does <u>not</u> submit the documentation within the specified period of time,	<ul style="list-style-type: none"> <li>• Notify the SMO for contract-compliance action.</li> <li>• Go to Step 6.</li> </ul>

6. Record the presence or absence ("Yes" or "No") of each item, as appropriate, in Section II (Completeness Check) of the Data-Validation Cover Sheet.
  - A. If the RRES-R Program does not request the TICs, record "n/a" (for "not analyzed") in blocks 9 and 10 of the completeness section of the Data-Validation Cover Sheet.
  - B. Also indicate any samples whose data are missing from the data-record package.
7. Photocopy the following:
  - Form 1, the results report from the analytical laboratory that will be used during the validation process
  - The chain of custody form

**Note:** Do not record data-validation qualifiers and reason codes on the original form (Form 1).

**Note:** Each page of the Form 1 must be initialed and dated by the validator; these initials and date must be present even if the validator accepts laboratory qualification.

8. Submit the photocopies of the items listed in Step 7 as attachments to the completed Data-Validation Checklist(s).
9. Go to Section 6.2, "Verifying the Holding Time."

## 6.2 Verifying the Holding Time

**Table 6.2-1**  
**Holding Time Acceptance Criteria**

Sample Matrix	Holding Time (days)
Soil (including ENCORE)	14
Water	7 if not acidified; 14 if acidified
Applicable storage conditions are listed in the current SOW for analytical services.	

**Note:** For water and low-level soil samples, extractions are conducted at the time of analysis, and no analysis holding time applies. For mid- and high-level soil analyses, a methanol extraction is used, and samples have to be extracted following the criteria in Table 6.2-1 and analyzed within 40 days after extraction to meet the analytical holding time acceptance criteria.

1. IF...	THEN...
<u>All</u> the samples were extracted and analyzed within the appropriate holding time (Table 6.2-1),	<ul style="list-style-type: none"> <li>Record "No" on lines 1 and 2 of the VOC Data-Validation Checklist (Attachment D).</li> <li>Go to Section 6.3, "Verifying the Instrument Performance Check."</li> </ul>
<u>Any</u> samples were <u>not</u> extracted and analyzed within the appropriate holding time (Table 6.2-1),	<ul style="list-style-type: none"> <li>Calculate the number of days the holding time requirement was exceeded.</li> <li>Go to Step 2.</li> </ul>

2. <b>IF the holding time criteria is...</b>	<b>THEN...</b>
Exceeded by 2 times,	<ul style="list-style-type: none"> <li>Record "No" on line 1 and "Yes" on line 2 of the VOC data validation Checklist.</li> <li>Qualify all the affected analytes as rejected (R, V9a) on Form 1.</li> <li>Go to Section 6.3, "Verifying the Instrument Performance Check."</li> </ul>
<u>Not</u> exceeded by 2 times,	<ul style="list-style-type: none"> <li>Record "Yes" on line 1 and "No" on line 2 of the VOC data validation Checklist</li> <li>Qualify the detected analytes as estimated (J, V9) and the nondetected analytes as estimated (UJ, V9) on Form 1.</li> <li>Go to Section 6.3, "Verifying the Instrument Performance Check."</li> </ul>

### 6.3 Verifying the Instrument Performance Check

<b>IF the bromofluorobenzene (BFB) instrument check...</b>	<b>THEN...</b>
Was completed within 12 h of the corresponding sample and calibration analyses <u>and</u> passed the acceptance criteria,	<ul style="list-style-type: none"> <li>Record "No" on line 3 of the VOC Data-Validation Checklist.</li> <li>Go to Section 6.4, "Verifying the Initial Calibration."</li> </ul>
Was <u>not</u> completed within 12 h of the corresponding sample analyses <u>or</u> failed acceptance criteria,	<ul style="list-style-type: none"> <li>Record "Yes" on line 3 of the VOC Data-Validation Checklist.</li> <li>Qualify the affected analytes as rejected (R, V16a) on the individual sample Form 1.</li> <li>Go to Section 6.4, "Verifying the Initial Calibration."</li> </ul>

#### 6.4 Verifying the Initial Calibration

1.	<b>IF the initial calibration information is...</b>	<b>THEN...</b>
	Present,	<ul style="list-style-type: none"><li>• Record "No" on line 4 of the VOC Data-Validation Checklist.</li><li>• Go to Step 2.</li></ul>
	Missing,	<ul style="list-style-type: none"><li>• Record "Yes" on line 4 of the VOC Data-Validation Checklist.</li><li>• Contact the analytical laboratory and the SMO for the missing information (see Section 6.1 -4).</li><li>• If the laboratory is unable to provide the missing information, qualify all the results as rejected (R, V16) on the individual sample Form 1.</li><li>• Go to Step 2.</li></ul>

2.	<b>IF the initial calibration information...</b>	<b>THEN...</b>
	Has five calibration points and a standard at or below the reporting limit,	<ul style="list-style-type: none"><li>• Record "No" on line 5 of the VOC Data-Validation Checklist.</li><li>• Go to Step 3.</li></ul>
	<u>Does not</u> have five calibration points or a standard at or below the reporting limit,	<ul style="list-style-type: none"><li>• Record "Yes" on line 5 of the VOC Data-Validation Checklist.</li><li>• Qualify the affected analytes as rejected (R, V7) on the individual sample Form 1.</li><li>• Go to Step 3.</li></ul>

3. <b>IF the %RSD...</b>	<b>THEN...</b>
For <u>each</u> analyte is = to 30%,	<ul style="list-style-type: none"> <li>Record "No" on line 6 of the VOC Data-Validation Checklist.</li> <li>Go to Step 4.</li> </ul> <p><b>Note:</b> If the %RSD is not used, the correlation coefficient must be greater than 0.995.</p>
For <u>any</u> analyte is > 30% <u>or</u> the correlation coefficient is < 0.995,	<ul style="list-style-type: none"> <li>Record "Yes" on line 6 of the VOC Data-Validation Checklist.</li> <li>Qualify the detected the analytes as estimated (J, V7a) and the nondetected analytes as estimated (UJ, V7a) on the individual sample Form 1.</li> <li>Go to Step 4.</li> </ul>

4. <b>IF the minimum RRF is...</b>	<b>THEN...</b>
= 0.05 for <u>each</u> analyte,	<ul style="list-style-type: none"> <li>Record "No" on line 7 of the VOC Data-Validation Checklist.</li> <li>Go to Section 6.5, "Verifying the Continuing Calibration."</li> </ul>
< the 0.05 for <u>any</u> analyte,	<ul style="list-style-type: none"> <li>Record "Yes" on line 7 on the VOC Data-Validation Checklist</li> <li>Qualify the detected analytes as estimated (J, V7b) and nondetects as rejected (R, V7b) on the individual sample Form 1.</li> <li>Go to Section 6.5, "Verifying the Continuing Calibration."</li> </ul>

## 6.5 Verifying the Continuing Calibration

1. <b>IF the continuing calibration information is...</b>	<b>THEN...</b>
Present,	<ul style="list-style-type: none"> <li>Record "No" on line 8 of the VOC Data-Validation Checklist.</li> <li>Go to Step 2.</li> </ul>

1. <b>IF the continuing calibration information is...</b>	<b>THEN...</b>
Missing,	<ul style="list-style-type: none"> <li>Record "Yes" on line 8 of the VOC Data-Validation Checklist.</li> <li>Contact the analytical laboratory and the SMO for the missing information (see Section 6.1 -4).</li> <li>If the laboratory is unable to provide the missing information, qualify all results as rejected (R, V16) on the individual sample Form 1.</li> <li>Go to Step 2.</li> </ul>

2. <b>IF the %D for...</b>	<b>THEN...</b>
<u>Each</u> analyte is = 25%,	<ul style="list-style-type: none"> <li>Record "No" on line 9 of the VOC Data-Validation Checklist.</li> <li>Go to Step 3.</li> </ul>
<u>Any</u> analyte is > 25%,	<ul style="list-style-type: none"> <li>Record "Yes" on line 9 of the VOC Data-Validation Checklist</li> <li>Qualify the detected analytes as estimated (J, V7a) and nondetected analytes as estimated (UJ, V7a) on the individual sample Form 1.</li> <li>Go to Step 3.</li> </ul>

3. <b>IF the minimum RRF is...</b>	<b>THEN...</b>
= 0.05 for each analyte,	<ul style="list-style-type: none"> <li>Record "No" on line 10 of the VOC Data-Validation Checklist.</li> <li>Go to Section 6.6, "Verifying the Method-Blank Results."</li> </ul>



3. <b>IF the minimum RRF is...</b>	<b>THEN...</b>
< 0.05 for any analyte,	<ul style="list-style-type: none"> <li>Record "Yes" on line 10 of the VOC Data-Validation Checklist.</li> <li>Qualify the detected analytes as estimated (J, V7b) and nondetects as rejected (R, V7b) on the individual sample Form 1.</li> <li>Go to Section 6.6, "Verifying the Method-Blank Results."</li> </ul>

## 6.6 Verifying the Method-Blank Results

**Note:** The data validator must compare the method-blank results to the contractually required estimated quantitation limits (EQLs) for each analytical batch.

**Note:** An analytical batch is any group of field samples and QC samples analyzed within 12 h of the BFB analysis

1. <b>IF the method blank information is...</b>	<b>THEN...</b>
Present,	<ul style="list-style-type: none"> <li>Record "No" on line 11 of the VOC Data-Validation Checklist.</li> <li>Go to Step 2.</li> </ul>
Missing,	<ul style="list-style-type: none"> <li>Record "Yes" on line 11 of the VOC Data-Validation Checklist.</li> <li>Contact the analytical laboratory and the SMO for the missing information (see Section 6.1 -4).</li> <li>If the laboratory is unable to provide the missing information, qualify all results as rejected (R, V4b) on the individual sample Form 1.</li> <li>Go to Step 2.</li> </ul>

2.	<b>IF the method blank has...</b>	<b>THEN...</b>
	<u>No</u> contamination,	<ul style="list-style-type: none"> <li>Record "No" on lines 12 and 13 of the VOC Data-Validation Checklist</li> <li>Go to Section 6.7, "Verifying the Internal Standards."</li> </ul>
	Contamination,	<ul style="list-style-type: none"> <li>Go to Step 3.</li> </ul>

3.	<b>IF the concentrations of any analyte in a sample is...</b>	<b>THEN..</b>
	= 5 times the concentration of that analyte in the corresponding method blank (10 times for common laboratory contaminants),	<ul style="list-style-type: none"> <li>Record "Yes" on line 12 of the VOC Data-Validation Checklist</li> <li>Qualify the detected analytes as undetected (U, V4) on the individual sample Form 1.</li> <li>Go to Section 6.7, "Verifying the Internal Standards."</li> </ul>
	> 5 times the concentration of that analyte in the corresponding method blank (10 times for common laboratory contaminants),	<ul style="list-style-type: none"> <li>Record "Yes" on line 13 of the VOC Data-Validation Checklist</li> <li>Qualify the detected analytes as estimated (J,V4a) on the individual sample Form 1.</li> <li>Go to Section 6.7, "Verifying the Internal Standards."</li> </ul>

**Note:** Check the concentrations of common laboratory contaminants in the diluted samples. If the concentration (divided by the dilution factor) is within 10 times the concentration of the blank, and the sample was NOT diluted for that analyte, use professional judgment to apply the qualification criteria listed in this section.

## 6.7 Verifying Internal Standards

**Note:** At least three of the four approved IS compounds listed in Table 6.7-1 are required for each sample. Therefore, retention times and area counts must be reported for at least three IS compounds in all the samples.

**Table 6.7-1  
RRES-R Program-Approved VOC Internal Standards**

IS Number	IS Name
1	Pentafluorobenzene
2	1,4-difluorobenzene
3	Chlorobenzene-d5
4	1,4-dichlorobenzene-d4

1.	<b>IF the IS information is...</b>	<b>THEN...</b>
	Present,	<ul style="list-style-type: none"> <li>Go to Step 2.</li> </ul>
	Missing,	<ul style="list-style-type: none"> <li>Record "Yes" on line 14 of the VOC Data-Validation Checklist.</li> <li>Contact the analytical laboratory and the SMO for the missing information (see Section 6.1 -4).</li> <li>If the laboratory cannot provide the missing information, qualify all the results as rejected (R, V2a).</li> <li>Go to Step 2.</li> </ul>
2.	<b>IF the IS area for...</b>	<b>THEN...</b>
	<u>All</u> of the three required IS compounds in the daily CCV are > 50% <u>or</u> < than 200% of the previous 12-h CCV,	<ul style="list-style-type: none"> <li>Record "No" on line 15 of the VOC Data-Validation Checklist.</li> <li>Go to Step 3.</li> </ul>
	<u>Any</u> of the three required IS compounds in the daily CCV are < 50% <u>or</u> > 200% of the previous 12-h CCV,	<ul style="list-style-type: none"> <li>Record "Yes" on line 15 of the VOC Data-Validation Checklist.</li> <li>Qualify the detected analytes as estimated (J, V1) and the nondetected analytes as estimated (UJ, V1) on the individual sample Form 1.</li> <li>Go to Step 3.</li> </ul>

3. <b>IF the IS retention times for any of the three required IS compounds have...</b>	<b>THEN...</b>
<u>Not</u> shifted by more than 30 s from the previous 12-h CCV,	<ul style="list-style-type: none"> <li>Record "No" on line 16 of the VOC Data-Validation Checklist .</li> <li>Go to Step 4.</li> </ul>
Shifted by more than 30 s from the previous 12-h CCV,	<ul style="list-style-type: none"> <li>Record "Yes" on line 16 of the VOC Data-Validation Checklist.</li> <li>Qualify all the affected analytes as estimated (J, V0/UJ, V0) on the individual sample Form 1.</li> <li>Go to Step 4.</li> </ul>

4. <b>IF the IS area for...</b>	<b>THEN...</b>
<u>All</u> of the three required IS compounds in a sample are < 200% of the previous 12-h CCV,	<ul style="list-style-type: none"> <li>Record "No" on line 17 of the VOC Data-Validation Checklist.</li> <li>Go to Step 5.</li> </ul>
<u>Any</u> of the three required IS compounds in a sample are > 200% of the previous 12-h CCV,	<ul style="list-style-type: none"> <li>Record "Yes" on line 17 of the VOC Data-Validation Checklist.</li> <li>Qualify all the detected analytes as estimated (J, V1a) on the individual sample Form 1.</li> <li>Go to Step 5.</li> </ul>

5. <b>IF the IS areas for...</b>	<b>THEN...</b>
<u>All</u> of the three required IS compounds in a sample are > 50% of the previous 12-h CCV,	<ul style="list-style-type: none"> <li>Record "No" on lines 18 and 19 of the VOC Data-Validation Checklist.</li> <li>Go to Section 6.8, "Verifying the Surrogate Recoveries."</li> </ul>
<u>Any</u> of the three required IS compounds in a sample are < 50% but = 10% of the previous 12-h CCV,	<ul style="list-style-type: none"> <li>Record "Yes" on line 18 of the VOC Data-Validation Checklist.</li> <li>Qualify the detected analytes as estimated (J, V1a) and the nondetected analytes as estimated (UJ, V1a) on the individual sample Form 1.</li> <li>Go to Step 6.</li> </ul>

6.	<b>IF the IS area for...</b>	<b>THEN...</b>
	<u>All</u> of the three required IS compounds in a sample are > 10% of the previous 12-h CCV,	<ul style="list-style-type: none"> <li>Record "No" on line 19 of the VOC Data-Validation Checklist</li> <li>Go to Section 6.8, "Verifying the Surrogate Recoveries."</li> </ul>
	<u>Any</u> of the three required IS compounds in a sample are < 10% of the previous 12-h CCV,	<ul style="list-style-type: none"> <li>Record "Yes" on line 19 of the VOC Data-Validation Checklist</li> <li>Qualify the detected analytes as estimated (J, V2) and the nondetected analytes as rejected (R, V2) on the individual sample Form 1.</li> <li>Go to Section 6.8, "Verifying the Surrogate Recoveries."</li> </ul>

## 6.8 Verifying the Surrogate Recoveries

**Note:** Surrogate percent recovery (%R) values that are outside the acceptance range listed in Table 6.7-1 as a result of sample dilution used to render target analytes quantifiable are not subject to the validation acceptance criteria presented in this section.

**Table 6.8-1**  
**VOC Surrogates and Recovery Acceptance Ranges**

Surrogate	Soil Matrix Acceptance Range (%R)	Water Matrix Acceptance Range (%R)
toluene-d8	81–117	88–110
4-bromofluorobenzene	74–121	86–115
1,2-dichloroethane-d4	50–120	80–120
dibromofluoromethane	80–120	86–118

1.	<b>IF the surrogate information is...</b>	<b>THEN...</b>
	Present,	<ul style="list-style-type: none"> <li>Go to Step 2.</li> </ul>

1.	<b>IF the surrogate information is...</b>	<b>THEN...</b>
	Missing,	<ul style="list-style-type: none"> <li>Record "Yes" on line 20 of the VOC Data-Validation Checklist.</li> <li>Contact the analytical laboratory and the SMO for the missing information (see Section 6.1 -4).</li> <li>If the laboratory is unable to provide the missing information, qualify all results as rejected (R, V3f), on the individual sample Form 1.</li> <li>Go to Step 2.</li> </ul>
2.	<b>IF...</b>	<b>THEN...</b>
	<u>All</u> surrogate %R in a sample is < the UAL,	<ul style="list-style-type: none"> <li>Record "No" on line 21 of the VOC Data-Validation Checklist.</li> <li>Go to Step 3.</li> </ul>
	<u>Any</u> surrogate %R in a sample is > the UAL,	<ul style="list-style-type: none"> <li>Record "Yes" on line 21 of the VOC Data-Validation Checklist</li> <li>Qualify all the detected analytes as estimated with a potential positive bias (J+, V3) on the individual sample Form 1.</li> <li>Go to Step 3.</li> </ul>
3.	<b>IF...</b>	<b>THEN...</b>
	<u>All</u> surrogate %R in a sample is > the lower acceptable limit (LAL),	<ul style="list-style-type: none"> <li>Record "No" on line 22 of the VOC Data-Validation Checklist</li> <li>Go to Section 6.9, "Verifying the Laboratory Control Sample Recoveries."</li> </ul>

3. <b>IF...</b>	<b>THEN...</b>
<u>Any</u> surrogate %R in a sample is < the LAL but = 10%,	<ul style="list-style-type: none"> <li>Record "Yes" on line 22 of the VOC Data-Validation Checklist</li> <li>Qualify the detected analytes as estimated with a potential negative bias (J-, V3a) and the nondetected analytes as estimated (UJ, V3c) on the individual sample Form 1.</li> <li>Go to Step 4.</li> </ul>

4. <b>IF...</b>	<b>THEN...</b>
<u>All</u> surrogate %R in a sample is > 10%,	<ul style="list-style-type: none"> <li>Record "No" on line 23 of the VOC Data-Validation Checklist.</li> <li>Go to Step 5.</li> </ul>
<u>Any</u> surrogate %R in a sample is < 10%,	<ul style="list-style-type: none"> <li>Record "Yes" on line 23 of the VOC Data-Validation Checklist.</li> <li>Qualify the detected analytes as estimated with a potential negative bias (J-, V3b) and the nondetected analytes as rejected (R, V3d) on the individual sample Form 1.</li> <li>Go to Step 5.</li> </ul>

5. <b>IF...</b>	<b>THEN...</b>
<u>No</u> surrogate %R in a sample is < the LAL <u>or no</u> surrogate %R is > the UAL,	<ul style="list-style-type: none"> <li>Record "No" on line 24 of the VOC Data-Validation Checklist.</li> <li>Go to Section 6.9, "Verifying the Laboratory Control Sample Recoveries."</li> </ul>

5. <b>IF...</b>	<b>THEN...</b>
<u>One</u> surrogate %R in a sample is < the LAL and any other surrogate %R is > the UAL,	<ul style="list-style-type: none"> <li>Record "Yes" on line 24 of the VOC Data-Validation Checklist.</li> <li>Qualify the detected analytes as estimated (J, V3e) and the nondetected analytes as estimated (UJ, V3e) on the individual sample Form 1.</li> <li>Go to Section 6.9, "Verifying the Laboratory Control Sample Recoveries."</li> </ul>

## 6.9 Verifying the Laboratory Control Sample Recoveries

**Note:** The laboratory may perform either a full analyte LCS or a short list LCS using the EPA-CLP matrix spike list.

1. <b>IF the LCS information is...</b>	<b>THEN...</b>
Present,	<ul style="list-style-type: none"> <li>Record "No" on line 25 of the VOC Data-Validation Checklist.</li> <li>Go to Step 2.</li> </ul>
Missing,	<ul style="list-style-type: none"> <li>Record "Yes" on line 25 of the VOC Data-Validation Checklist.</li> <li>Contact the analytical laboratory and the SMO for the missing information (see Section 6.1 -4).</li> <li>If the laboratory is unable to provide the missing information, qualify all the results as rejected (R, V12) on the individual sample Form 1.</li> <li>Go to Step 2.</li> </ul>

2. <b>IF...</b>	<b>THEN..</b>
<u>All</u> of the LCS analyte %R are < the UAL,	<ul style="list-style-type: none"> <li>Record "No" on line 26 of the VOC Data-Validation Checklist</li> <li>Go to Step 3.</li> </ul>



2.	<b>IF...</b>	<b>THEN..</b>
	<u>Any</u> LCS analyte %R are > the UAL,	<ul style="list-style-type: none"> <li>Record "Yes" on line 26 of the VOC Data-Validation Checklist</li> <li>Qualify all the positive analytes as estimated with a potential positive bias (J+, V12d) on the individual sample Form 1.</li> <li>Go to Step 3</li> </ul>
3.	<b>IF...</b>	<b>THEN...</b>
	<u>All</u> of the LCS analyte %R are > the LAL,	<ul style="list-style-type: none"> <li>Record "No" on lines 27 and 28 of the VOC Data-Validation Checklist.</li> <li>Go to Section 6.10, "Verifying the Mass Spectra."</li> </ul>
	<u>Any</u> of the LCS analyte %R are < the LAL, but are = 10%,	<ul style="list-style-type: none"> <li>Record "Yes" on line 27 of the VOC Data-Validation Checklist.</li> <li>Qualify the detected analytes as estimated with a potential negative bias (J-, V12b) and the nondetected analytes as estimated (UJ, V12c) on the individual sample Form 1.</li> <li>Go to Step 4.</li> </ul>
4.	<b>IF...</b>	<b>THEN..</b>
	<u>All</u> LCS analyte %R are > 10%	<ul style="list-style-type: none"> <li>Record "No" on line 28 of the VOC Data-Validation Checklist.</li> <li>Go to Section 6.10, "Verifying the Mass Spectra."</li> </ul>
	<u>Any</u> LCS analyte %R are < 10%,	<ul style="list-style-type: none"> <li>Record "Yes" on line 28 of the VOC Data-Validation Checklist.</li> <li>Qualify the detected analytes as estimated with a potential negative bias (J-, V12a) and the nondetected analytes as rejected (R,V12a) on the individual sample Form 1.</li> <li>Go to Section 6.10, "Verifying the Mass Spectra."</li> </ul>

## 6.10 Verifying the Mass Spectra

1. IF...	THEN...
<u>All</u> of the required mass spectra are <u>present</u> ,	<ul style="list-style-type: none"> <li>Record "No" on line 29 of the VOC Data-Validation Checklist.</li> <li>Go to Step 2.</li> </ul>
<u>Any</u> of the required mass spectra are <u>missing</u> ,	<ul style="list-style-type: none"> <li>Record "Yes" on line 29 of the VOC Data-Validation Checklist.</li> <li>Contact the analytical laboratory and the SMO for the missing information (see Section 6.1 -4).</li> <li>If the laboratory is unable to provide the missing information, qualify all the affected results as rejected (R, V8a), on the individual sample Form 1.</li> <li>Go to Step 2.</li> </ul>

2. IF...	THEN...
<u>All</u> mass spectra meet method specifications,	<ul style="list-style-type: none"> <li>Record "No" on line 30 of the VOC Data-Validation Checklist, and</li> <li>Go to Section 6.11, "Verifying the Tentatively Identified Compounds."</li> </ul>
<u>Any</u> mass spectra do <u>not</u> meet method specifications,	<ul style="list-style-type: none"> <li>Record "Yes" on line 30 of the VOC Data-Validation Checklist and</li> <li>Qualify all the detected analytes as undetected (U, V8) on the individual sample Form 1.</li> <li>Go to Section 6.11, "Verifying the Tentatively Identified Compounds."</li> </ul>

## 6.11 Verifying the Tentatively Identified Compounds

**Note:** If the order code contains "N" as the last letter, the "N" indicates that TICs were *not* requested.

IF the TIC information...	THEN...
Is <u>present</u> <u>or</u> was not requested,	<ul style="list-style-type: none"> <li>• Check “No” on line 31 of the VOC Data-Validation Checklist if TICs are present.</li> <li>• Check “n/a” if TICs were not requested.</li> <li>• Go to Section 6.12, “Verifying the Dilutions.”</li> </ul>
Was requested but is <u>missing</u> ,	<ul style="list-style-type: none"> <li>• Record “Yes” on line 31 of the VOC Data-Validation Checklist.</li> <li>• Contact the analytical laboratory and the SMO for the missing information (see Section 6.1 -4).</li> <li>• If the laboratory is unable to provide the missing information, qualify all results as rejected (R, V11) on the individual sample Form 1.</li> <li>• Go to Section 6.12, “Verifying the Dilutions.”</li> </ul>

#### 6.12 Verifying the Dilution

IF the sample was...	THEN...
<u>Not</u> diluted,	<ul style="list-style-type: none"> <li>• Record “No” on line 32 of the VOC Data-Validation Checklist.</li> <li>• Go to Section 6.13, “Identifying the Obvious Quality Deficiencies.”</li> </ul>

IF the sample was...	THEN...
Diluted and <u>no</u> target analytes were detected above the second lowest standard,	<ul style="list-style-type: none"> <li>Record "Yes" on line 32 of the VOC Data-Validation Checklist, and</li> <li>Contact the analytical laboratory and the SMO for the missing information (see Section 6.1 -4).</li> <li>If the laboratory cannot provide the missing information, qualify affected samples as rejected (R, V10) on the individual sample Form 1.</li> <li>If the laboratory can provide proof of matrix interference that was not removed by acceptable cleanup attempts, qualify all nondetected analytes as estimated (UJ, V10) on the individual sample Form 1.</li> <li>Go to Section 6.13, "Identifying the Obvious Quality Deficiencies."</li> </ul>

### 6.13 Identifying the Obvious Quality Deficiencies

1. IF the validator...	THEN...
Notifies <u>any</u> significant or obvious data quality deficiencies during the Data-Validation process,	<ul style="list-style-type: none"> <li>Record "Yes" on line 33 of the VOC Data-Validation Checklist.</li> <li>Contact the analytical laboratory and SMO, if necessary, to resolve the quality issue.</li> <li>Record the appropriate qualifier for the data based on the validator's best professional judgment, and apply reason code V19.</li> <li>Write a clear description of the flagged quality issue on the Data-Validation Cover Sheet.</li> <li>Go to Step 2.</li> </ul>
Sees there are <u>no</u> obvious data quality deficiencies other than those covered by this SOP,	<ul style="list-style-type: none"> <li>Record "No" on line 33 of the VOC Data-Validation Checklist.</li> <li>Go to Step 2.</li> </ul>

2. Ensure that the completed Data-Validation Cover Sheet is signed and dated.
3. Go to Section 6.14, “Assembling and Submitting the Data-Record Package.”

#### 6.14 Assembling and Submitting the Data-Record Package

1. Assemble the data-record package to include the following items in the following order:
  - The completed Data-Validation Cover Sheet.
  - The VOC Data-Validation Checklist (Sections 6.2 through 6.12 completed).
  - Photocopies of the form (Form 1) on which Data-Validation qualifiers and reason codes were recorded (assemble in order by sample identification number).
  - Photocopies of the data-record package chain of custody forms.
2. Attach the data-validation record package with the original data package and submit to the Facility Support Facility (FSF) in accordance with SOP-15.09.

## 7.0 LESSONS LEARNED

- 7.1 Before performing work described in this SOP, **RRES-R Personnel** should go to the Department of Energy Lessons Learned Information Services home page, located at <http://www.tis.eh.doe.gov/II/II.html>, and/or to the LANL Lessons Learned Resources web page, located at [http://www.lanl.gov/projects/lessons\\_learned/](http://www.lanl.gov/projects/lessons_learned/), and search for applicable lessons.
- 7.2 During work performance and/or after the completion of work activities, **RRES-R Personnel**, as appropriate, shall identify, document, and submit lessons learned in accordance with the LANL, Lessons Learned System located at [http://www.lanl.gov/projects/lessons\\_learned/](http://www.lanl.gov/projects/lessons_learned/).

## 8.0 RECORDS

Although completing this procedure generates no records, the items identified in Section 6.13 are part of the data-record package submitted to the RPF from the SMO in accordance with SOP-15.09.

## 9.0 REFERENCES

To properly implement this SOP, **RRES-R Personnel** should become familiar with the contents of the following documents located at [http://erinternal.lanl.gov/home\\_links/Library\\_proc.shtml](http://erinternal.lanl.gov/home_links/Library_proc.shtml):

- RRES-R Program Quality Management Plan
- EPA, "U.S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review," Publication 9240.1 -05, EPA-540/R-94/012, Office of Solid Waste and Emergency Response, Washington, DC. (EPA 1999, 66649) (October 1999)
- SOP-01.01, General Instructions for Field Investigations
- SOP-15.09, Chain of Custody for Analytical Data Packages
- LANL (Los Alamos National Laboratory), "Environmental Restoration Project Statement of Work for Analytical Services," Revision 2, Los Alamos National Laboratory document RFP Number 9-SX1-Q4257, (July 1995)
- QP-2.2, Personnel Orientation and Training
- QP-4.2, Standard Operating Procedure Development
- QP-5.7, Notebook Documentation for Environmental Restoration Technical Activities.

## 10.0 ATTACHMENTS

The **user** of this SOP may locate all forms associated with this procedure at <http://erinternal.lanl.gov/Quality/user/forms.asp>.

Attachment A: Volatile Organic Data-Validation Qualifier Flags, 1 page

Attachment B: Volatile Organic Data-Validation Reason Codes , 3 pages

Attachment C: Data-Validation Cover Sheet, 1 page

Attachment D: VOC Data-Validation Checklist, 1 page

Attachment E: List of Acronyms and Abbreviations, 1 page

## **Attachment A: Volatile Organic Data-Validation Qualifier Flags**

- R The analyte is classified “rejected.”
- J The analyte is classified “detected,” but the reported concentration value is expected to be more uncertain than usual.
- J+ The analyte is classified “detected,” but the reported concentration value is expected to be more uncertain than usual with a potential positive bias.
- U The analyte is classified “not detected.”
- UJ The analyte is classified “not detected” with an expectation that the reported result is more uncertain than usual.

## Attachment B: Volatile Organic Data-Validation Reason Codes

Code	Volatiles	Qualifier Nondetects	Qualifier Detects	Description	Comments
0	VO	See comments	See comments	Results for affected analytes are considered not detected (U)/rejected because the associated IS retention times have shifted by more than 30 s from the previous continuing calibration standard.	The validator must check the chromatogram profile to determine if any false positives or negatives exist. Qualify reported analytes not detected (U) if mass spectrum does not match. Reject analytes that are present but do not match mass spectrum and retention times have shifted by more than 30 s.
1	V1	UJ	J	Results/reporting limits for affected analytes are considered estimated (J)/(UJ) because associated IS area counts are less than 50% or greater than 200% of the previous continuing calibration standard.	Use when the validator compares the CCV to the previous day's CCV. The validator qualifies only analytes related to failing ISs.
1a	V1a	UJ	J	Results/reporting limits for affected analytes are considered estimated (J)/(UJ) because the associated IS area counts are less than 50% but greater than 10% of the previous continuing calibration standard.	Use when the validator compares the samples to the CCV in the analytical sequence. Validator qualifies only analytes related to failing ISs.
1b	V1b	N/A	J	Results for affected analytes are considered estimated (J) because the associated IS areas' counts are greater than 200% of the previous continuing calibration standard. The validator qualifies only analytes related to failing ISs.	
2	V2	R	J	Results/reporting limits for the affected analytes are considered estimated (J) for detected analytes/rejected for nondetected analytes because the associated IS area counts were less than 10% of the previous continuing calibration standard.	
2a	V2a	See comments	See comments	Required IS information is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
3	V3	N/A	J+	Results for the affected analytes are considered estimated with a potential positive bias (J+) because the sample surrogate recovery was greater than the UAL.	
3a	V3a	N/A	J-	Results for the affected analytes are considered estimated with a potential negative bias (J-) because the sample surrogate recovery was less than the LAL but greater than 10%.	Code is used only for detected analytes
3b	V3b	N/A	J-	Results for the affected analytes are considered estimated with a potential negative bias (J-) because the sample surrogate recovery was less than 10%.	Code is used only for detected analytes.
3c	V3c	UJ	N/A	Reporting limits for the affected analytes are considered estimated (UJ) because the sample surrogate recovery was less than the LAL but greater than or equal to 10%.	Code is used only for nondetected analytes.
3d	V3d	R	N/A	The reporting limits for the affected analytes are considered rejected because the sample surrogate recovery was less than 10%.	Code is used only for nondetected analytes.
3e	V3e	UJ for LAL	J+/UAL J-/LAL	Reporting limits/results for affected analytes are considered estimated (UJ)/estimated with a potential positive bias (J+)/estimated with a potential negative bias (J-) because at least one of the sample surrogates was greater than the UAL and one was less than the LAL.	
3f	V3f	See comments	See comments	Required surrogate information is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.



Code	Volatiles	Qualifier Nondetects	Qualifier Detects	Description	Comments
4	V4	N/A	U	Results for the affected analytes are considered not detected (U) because the associated sample concentration was less than 5 times/10 times the amount in the method blank.	Effective dilutions must be considered for common laboratory contaminants.
4a	V4a	N/A	J	Results for the affected analytes are considered estimated (J) because the associated sample concentration was greater than 5 times/10 times the amount in the method blank.	
4b	V4b	See comments	See comments	Required method blank or instrument blank documentation is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
7	V7	R	J	Results for affected analytes are considered rejected/estimated (J) because the associated analyte did not have a valid 5-point calibration and/or a standard at the reporting limit.	Qualify only affected analytes.
7a	V7a	UJ	J	Results/reporting limits for affected analytes are considered estimated (J)/estimated (UJ) because the associated %RSD/%D exceeded criteria in the initial or continuing calibration standards.	Qualify only affected analytes.
7b	V7b	R	J	Results/reporting limits for the affected analytes are considered estimated (J) for detects/rejected for nondetects because the associated RRF was less than 0.05.	Qualify only affected analytes.
8	V8	N/A	U	Results for the affected analytes are considered not detected (U) because the associated mass spectrum did not meet method specifications.	
8a	V8a	See comments	See comments	Mass spectrum documentation is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
9	V9	UJ	J-	Results/reporting limits for affected analytes are considered estimated with a potential negative bias (J-)/estimated (UJ) because the extraction/analytical holding time was exceeded by less than 2 times the published method holding time requirement.	
9a	V9a	R	R	Results for affected analytes are considered rejected because the sample analysis exceeded 2 times the method published holding time requirement.	
10	V10	See comments	See comments	Undetected results for affected analyte are considered estimated (UJ) or rejected because the laboratory diluted the sample for matrix interferences.	Qualify all results as rejected if the laboratory cannot provide proof of cleanup or matrix interferences. Qualify nondetected results as estimated if the laboratory can provide evidence of cleanup and/or matrix interferences not subject to acceptable cleanup methods.
11	V11	See comments	See comments	TICs are not reported but were requested by the RRES-R Program. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
12	V12	See comments	See comments	LCS documentation is missing. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
12a	V12a	R	J-	Results/reporting limits for affected analytes should be regarded as estimated with a potential negative bias (J-) for detects/rejected for nondetects because the associated LCS recovery was less than 10%.	Qualify all analytes quantitated by the same IS as the failing analyte.
12b	V12b	N/A	J-	Results for the affected analyte are considered estimated with a potential negative bias (J-) because the associated LCS recovery was less than the LAL but greater than 10%.	Qualify all analytes quantitated by the same IS as the failing analyte. This code is for detected analyses.

Code	Volatiles	Qualifier Nondetects	Qualifier Detects	Description	Comments
12c	V12c	UJ	N/A	Reporting limits for affected analyte are considered estimated (UJ) because the associated LCS recovery was less than the LAL but greater than 10%.	Qualify all analytes quantitated by the same IS as the failing analyte. This code is for nondetected analyses.
12d	V12d	N/A	J+	Results for affected analyte are considered estimated with a potential positive bias (J+) because the associated LCS recovery was greater than the UAL.	Qualify all analytes in that fraction (VOC) and/or all analytes quantitated by the same IS as the failing analyte.
15	V15			Results for the affected sample could not be analyzed because of insufficient sample volume or problems at the analytical laboratory.	
16	V16	See comments	See comments	Required calibration information is missing or samples were analyzed on an expired calibration. Validation cannot proceed without this information.	The package should be returned to the SMO, or the information should be requested from the laboratory.
16a	V16a	R	R	Results/reporting limits for affected analytes are rejected because the BFB instrument performance sample did not pass method-acceptance criteria.	
19	V19	See comments	See comments	Validator identified quality deficiencies in the reported data that require qualification. See the Data-Validation Cover Sheet for specific details.	Apply the appropriate qualifier to identify the effect of the quality deficiency on the reported data.

**Note:** The validator must notify the project manager and the SMO of any rejected data for potential nonpayment of services.

## Attachment C: Data-Validation Cover Sheet

☐ **Rejected Data**

### Section I

Request Number: \_\_\_\_\_ Validation Date: \_\_\_\_\_ Lab Code: \_\_\_\_\_

Contract Laboratory Name: \_\_\_\_\_ Validator: \_\_\_\_\_

Organization: \_\_\_\_\_

Analytical Suite (check all that apply):

<input type="checkbox"/> Volatile Organics	<input type="checkbox"/> High Explosives
<input type="checkbox"/> Semivolatile Organics	<input type="checkbox"/> Inorganics
<input type="checkbox"/> Organochlorine Pesticides/Polychlorinated Biphenyls	<input type="checkbox"/> Radiochemistry

Other (describe): \_\_\_\_\_

### Section II—Completeness Check

Yes	No	n/a	(check one)	Yes	No	n/a	(check one)
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	1. Chain-of-custody form(s)	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	6. Raw/BSS data
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2. Case narrative	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7. Quality control forms
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	3. Sample result forms	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	8. Quantitation reports
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	4. Sample chromatograms	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9. TICs forms
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	5. Standard chromatograms	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	10. TICs mass spectra

Comments/problems noted (include information about requests for further information submitted to the contract laboratory and agreed-upon date of resolution and contract laboratory point of contact):

(Attach additional comment sheets as necessary.)

Validator's signature: \_\_\_\_\_ Date: \_\_\_\_\_

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## Attachment D: VOC Data-Validation Checklist

Yes	No	n/a	(check one)	Assign qualifier listed below if criteria = Yes	
				Detected analyte	Undetected analyte
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	1. The holding time was >1 and =2 times the applicable holding time requirement.	J, V9	UJ, V9
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	2. The holding time was >2 times the applicable holding time requirement.	R, V9a	R, V9a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	3. The instrument performance check was not present or fails acceptance criteria.	R, V16a	R, V16a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	4. The initial calibration was not present.	R, V16	R, V16
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	5. The initial calibration did not have 5 calibration points and a low standard at the reporting limit.	R, V7	R, V7
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	6. The initial calibration analyte %RSD was >30% .	J, V7a	UJ, V7a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	7. The analyte minimum RRF was <0.05.	J, V7b	R, V7b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	8. The CCV was not present.	R, V16	R, V16
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	9. The continuing calibration analyte %D was >25%.	J, V7a	UJ, V7a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	10. The analyte minimum RRF was <0.05.	J, V7b	R, V7b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	11. The method blank was not reported.	R, V4b	R, V4b
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	12. The analyte detected in the method blank <u>and</u> sample result for analyte was < 5x/10x the amount in the blank.	U, V4	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	13. The analyte detected in the method blank <u>and</u> sample result for analyte was >5x/10x the amount in the method blank.	J, V4a	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	14. The IS information was not present.	R, V2a	R, V2a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	15. The IS areas in CCV were <50% or >200% of the previous days' IS areas.	J, V1	UJ, V1
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	16. The IS retention times had shifted by >30 s.	J, V0	UJ, V0
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	17. The sample IS areas were >200% of the daily CCV IS areas.	J, V1b	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	18. The sample IS areas were <50% but are =10% of the daily CCV IS areas.	J, V1a	UJ, V1a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	19. The sample IS areas were <10% of the daily CCV IS areas.	J, V2	R, V2
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	20. The surrogate information was not present.	R, V3f	R, V3f
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	21. The surrogate %R was >UAL.	J+, V3	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	22. The surrogate %R was <LAL but =10%.	J-, V3a	UJ, V3c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	23. The surrogate %R was <10%.	J-, V3b	R, V3d
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	24. The surrogate recoveries were (1) <LAL and (1) >UAL.	J, V3e	UJ, V3e
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	25. The LCS information was not present.	R, V12	R, V12
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	26. The LCS %R was >UAL.	J+, V12d	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	27. The LCS %R was <LAL but =10%.	J-, V12b	UJ, V12c
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	28. The LCS %R was <10%.	J-, V12a	R, V12a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	29. The mass spectral information was missing.	R, V8a	R, V8a
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	30. The mass spectrum did not meet method specifications.	U, V8	N/A
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	31. The TICs were requested but not reported.	R, V11	R, V11
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	32. The sample was diluted inappropriately.	N/A	U, V10
<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	33. Other obvious data-quality issues were identified.	__, V19	__, V19

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## Attachment E: List of Acronyms and Abbreviations

BFB	bromofluorobenzene	n/a	not analyzed
BSS	background subtracted spectra	%D	percent difference
CCV	continuing calibration verification	%R	percent recovery
CLP	Contract Laboratory Program	%RSD	percent relative standard deviation
COC	chain of custody	QC	quality control
EPA	U.S. Environmental Protection Agency	QP	quality procedure
EQL	estimated quantitation limit	RIC	restructured ion chromatogram
RRES-R	environmental restoration	RL	reporting limit
GC/MS	gas chromatography/mass spectrometry	RN	request number
FSF	field support facility	RPF	Records Processing Facility
HE	high explosive	RRF	relative response factor
IS	internal standard	RT	retention time
LAL	lower acceptance limit	SMO	Sample Management Office
LANL	Los Alamos National Laboratory	SOP	standard operating procedure
LCS	laboratory control sample	SOW	statement of work
MDL	method detection limit	TIC	tentatively identified compound
		UAL	upper acceptance limit
		VOC	volatile organic compound